

NISTIR 6527

Measurement Needs for Fire Safety: Proceedings of an International Workshop

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U.S. Department of Commerce

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Extractive methods

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SP Swedish National Testing and Research Institute

Presentation at NIST 2000-04-05



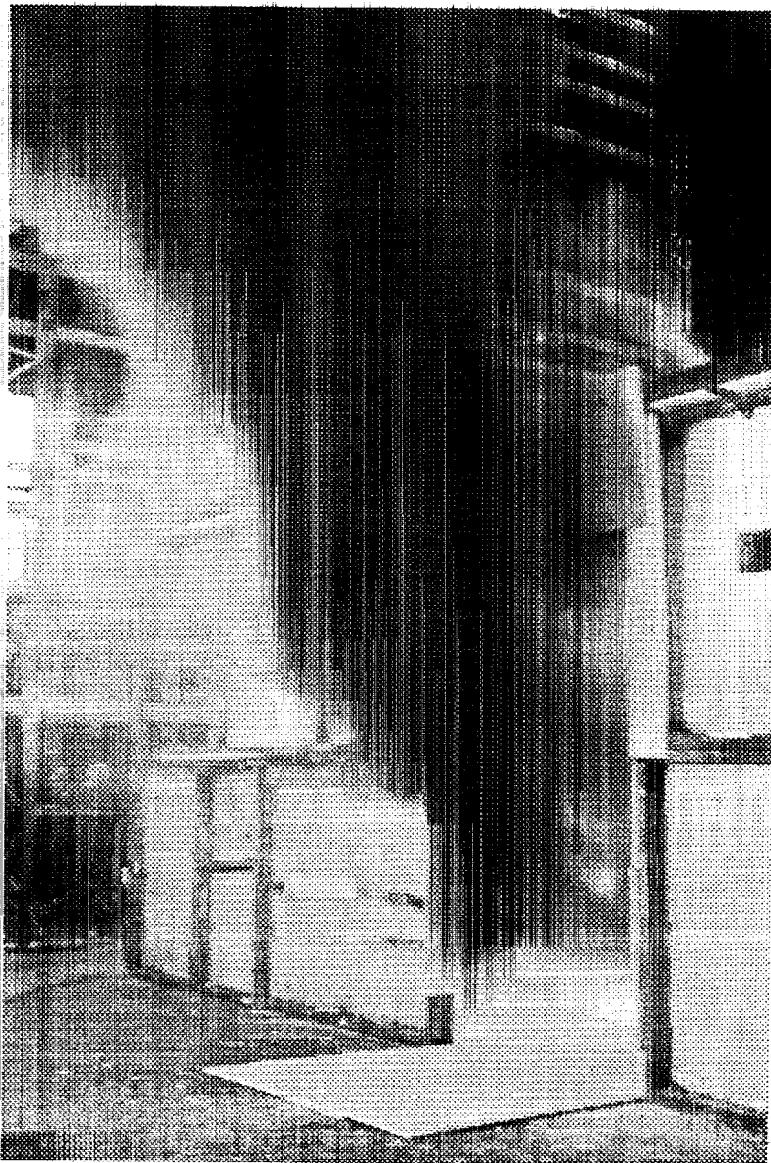
Why use extractive methods?

- More well-developed compared to optical methods,
e.g. Laser methods

Problems in using optical techniques:

- High particle loading
- Limited optical access
- Optical signal absorbed
- Large-scale applications

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Sampling

In the flames:

- Probe-induced disturbance
- Quenching needed
- Catalytic effects

In an opening:

- Non-uniform concentrations
- High concentrations

In a duct:

- More well-mixed
- Lower concentrations

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Measurement of Fire Gases

Fire Gases:

CO₂, CO, HBr, HCl, HF,

NO, NO₂, HCN, NH₃,

SO₂, COS, C₂H₂,

VOC, PAH, Dioxins

Measurement techniques:

- Sampling for single species
- Dedicated on-line analysers
- FTIR
- GC/MS

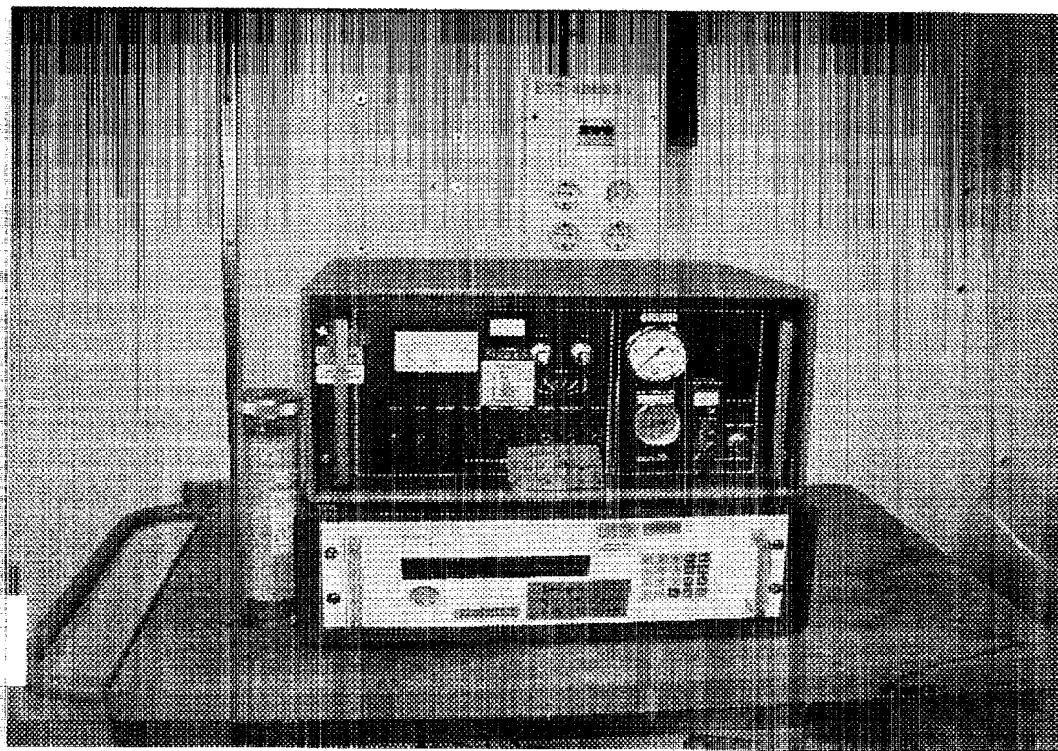
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Absorption solution

Gas specie	Absorption solution	Analysis method	Limit of detection (ppm (V/V))
Hydrogen chloride (HCl)	1.8 nM Na ₂ CO ₃ / 1.7 mM NaHCO ₃	Ion chromatography	0.15
Hydrogen cyanide (HCN)	0.5 M NaOH	UV spectroscopy	0.10
Ammonia (NH ₃)	5 mM H ₂ SO ₄	UV spectroscopy	0.15

(Sampling 2 l/min for 10 min)

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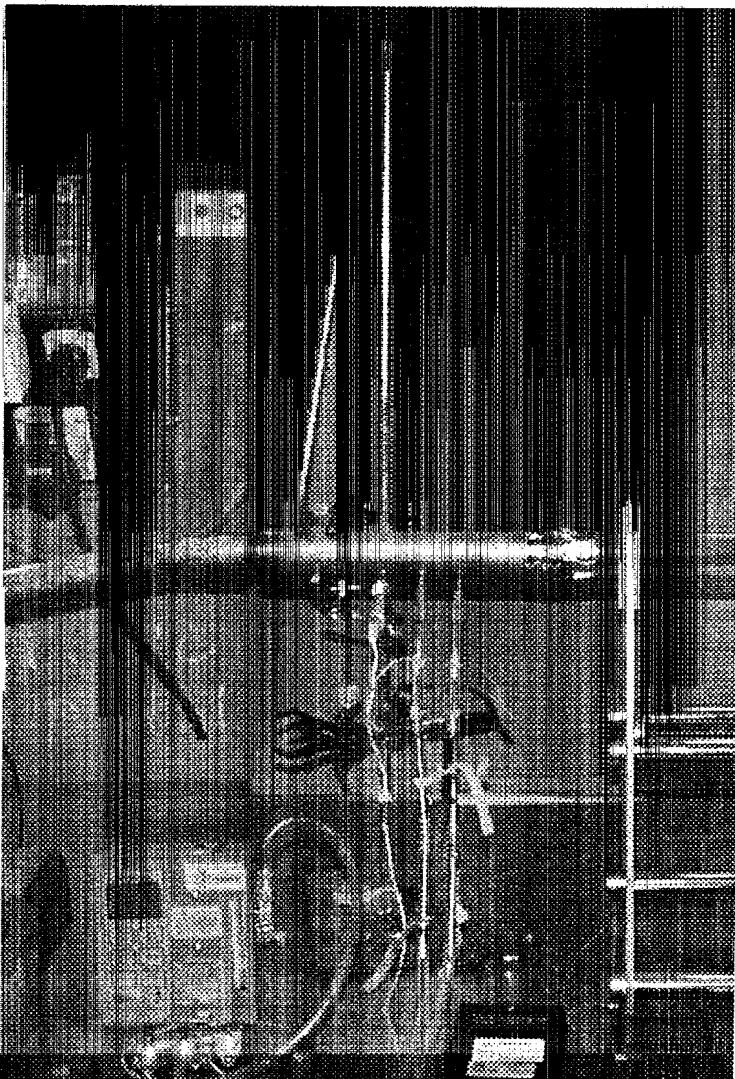


Dedicated on-line analysers

- Reliable
- Often fast (sec.)
- Broad conc. Ranges
(ppm to %)
- Often single species ...

Applications:

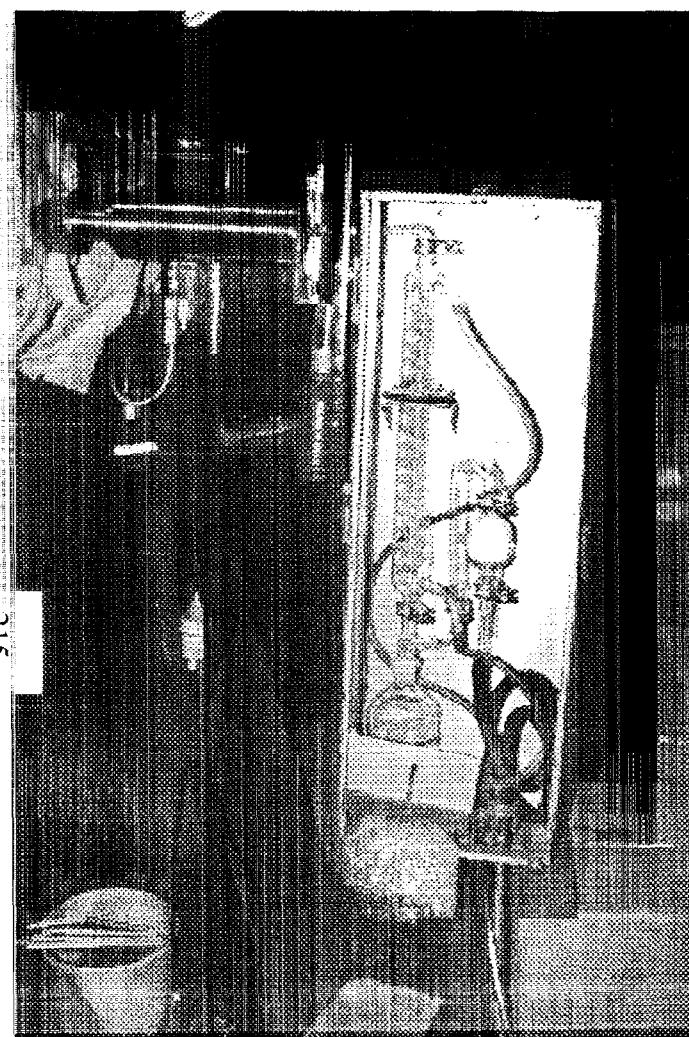
O_2 , CO_2/CO , NO_x ,
 SO_x , THC etc.



Adsorbent sampling I

- **Accumulative**
- **Low sampling flow**
- **Slow (minutes)**
- **Design for smaller organic species**
- **Thermal desorption**
- **~ 0.1 ppb Benzene, 1 min**

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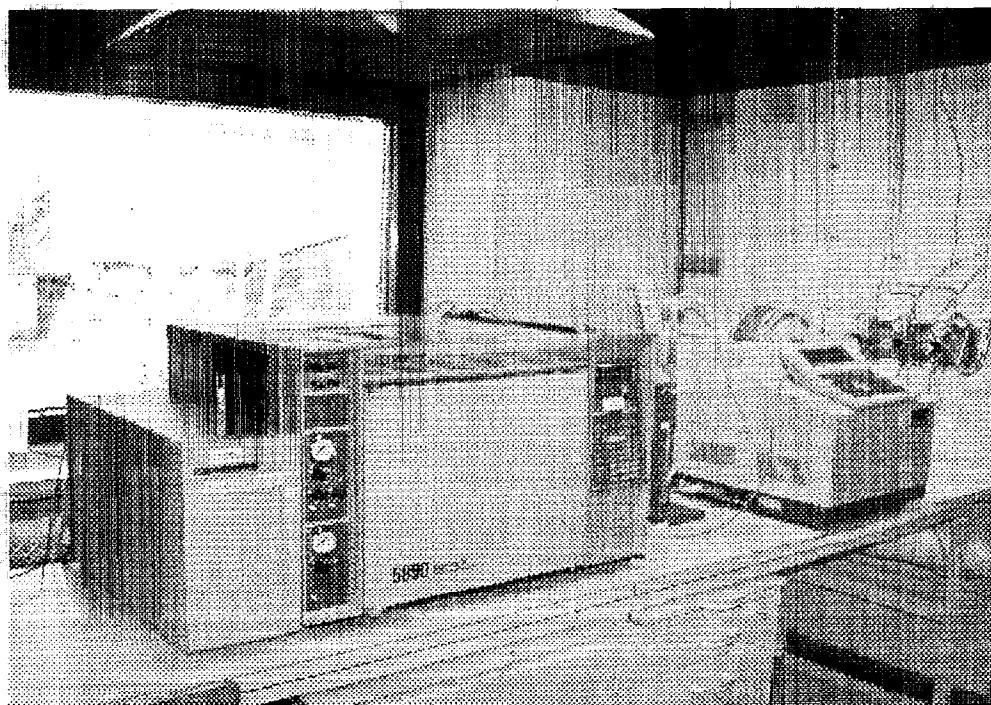


Adsorbent sampling II

- **Accumulative**
- **High sampling flow**
- **Slower (minutes ...)**
- **Design for larger organic species**
- **Solvent desorption**
- **~ 0.000001 ppb TCDD, 10 min**

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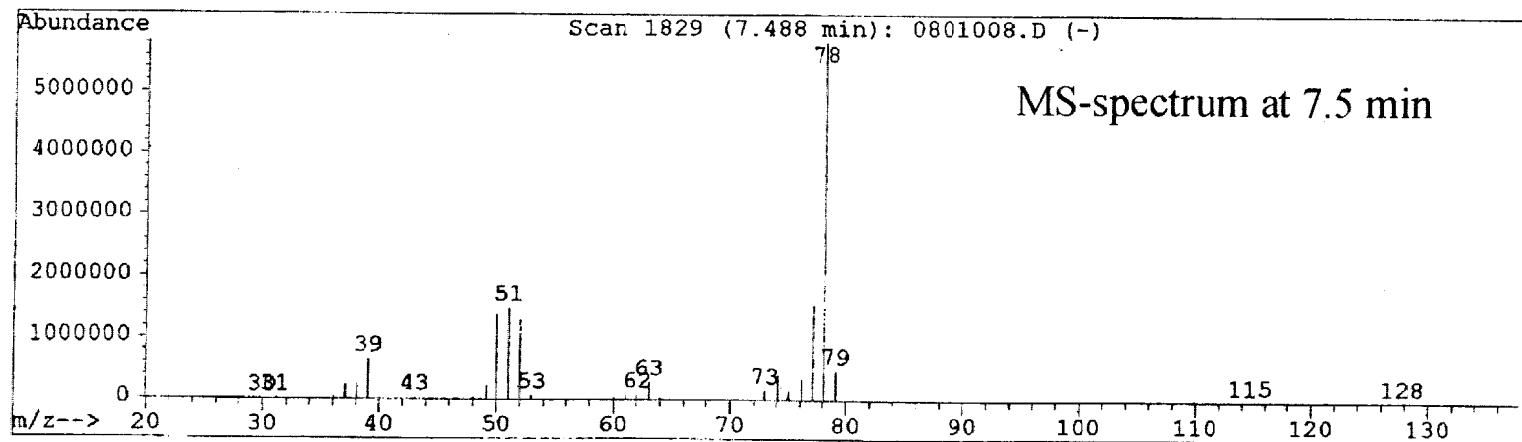
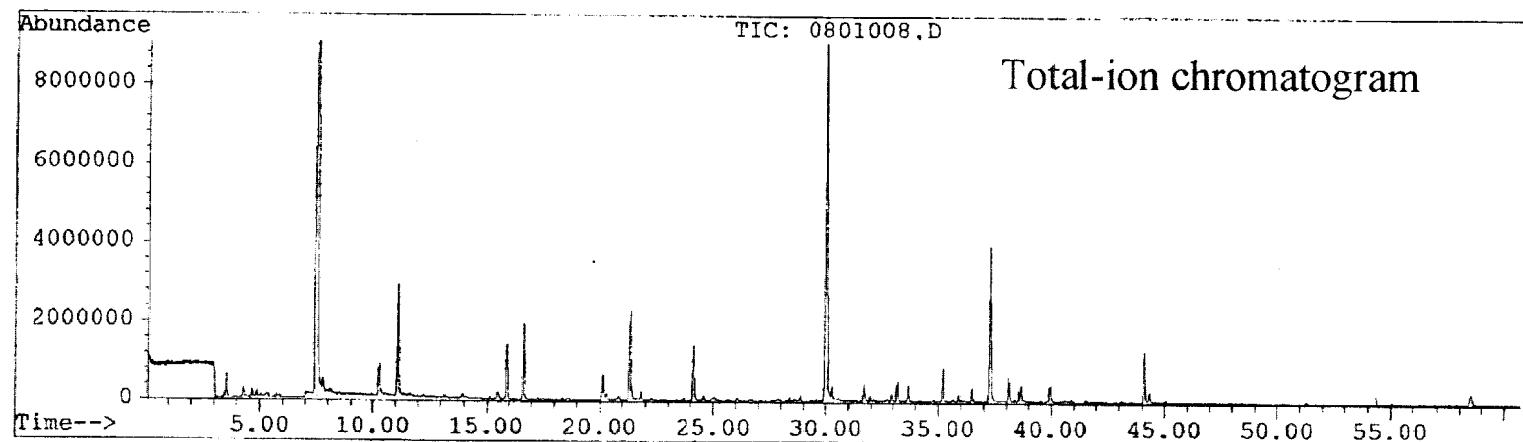
GC – MS/FID analysis



- Automatic thermal desorption
- Accumulation in cryogenic trap
- Separation on GC-column
- Identification MS
- Quantification FID
- Detection: ng

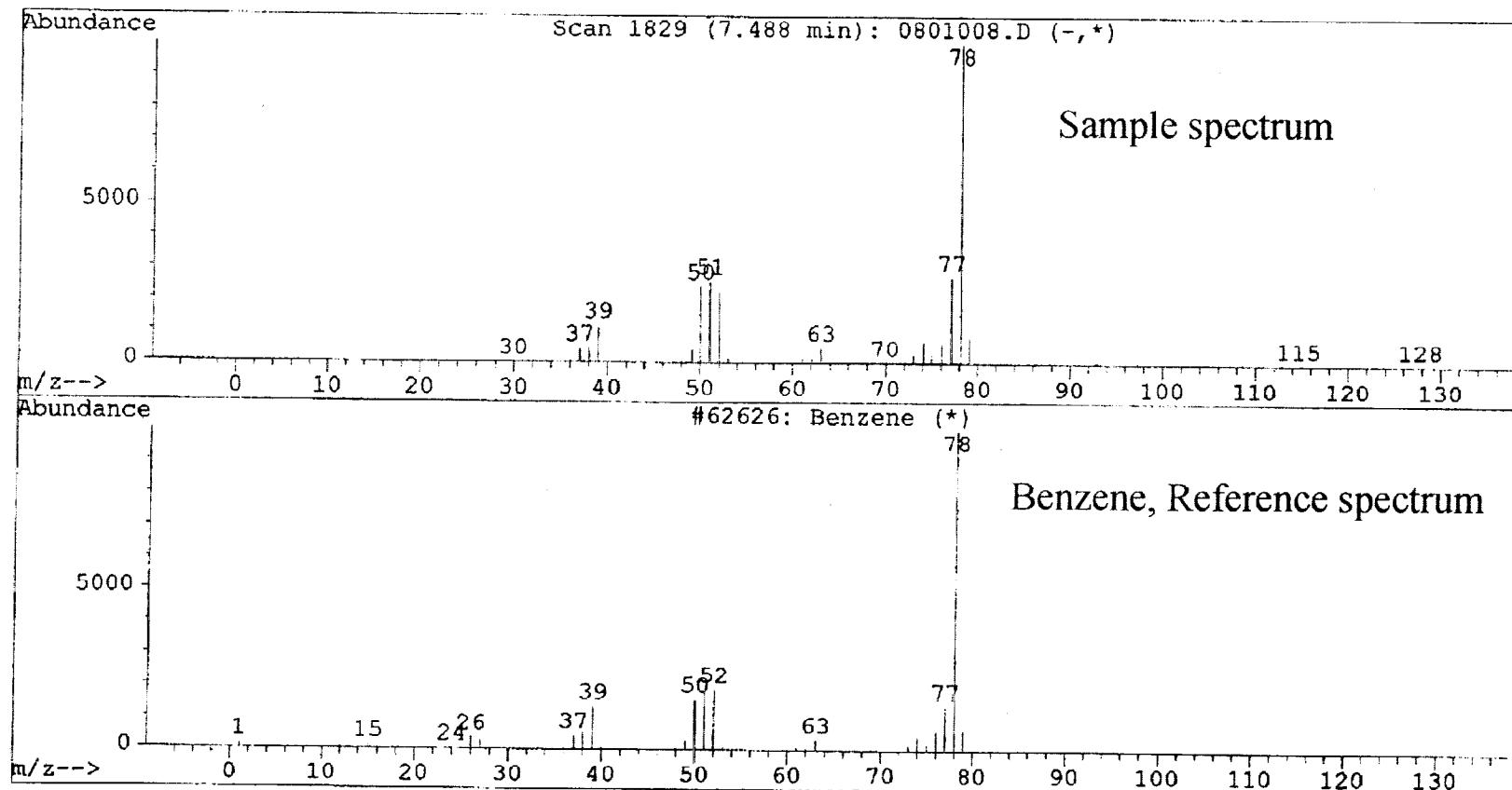
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MS analysis of fire test sample



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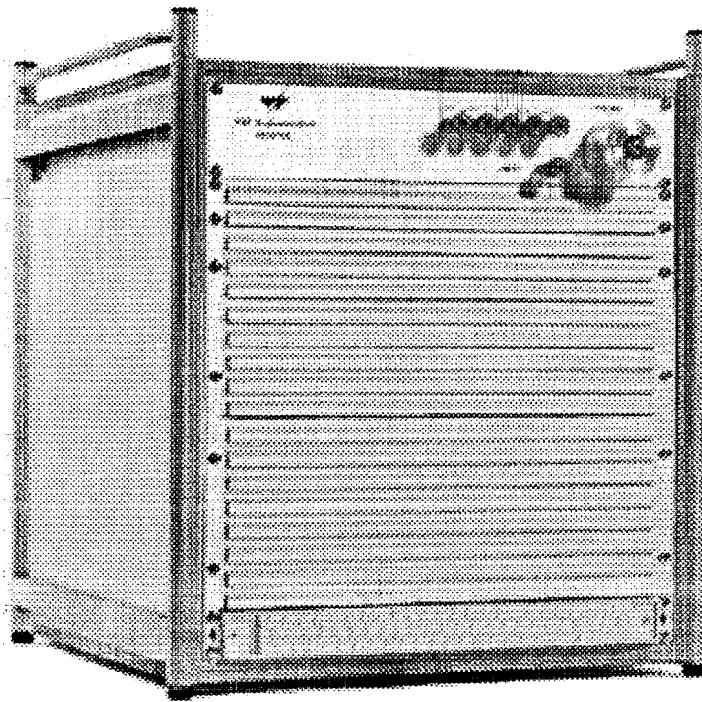
Identification from MS-spectra



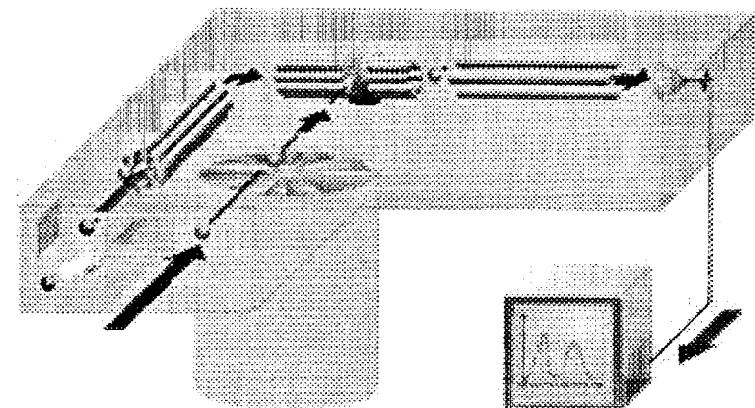
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On-line MS

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610 × 570 × 700 mm



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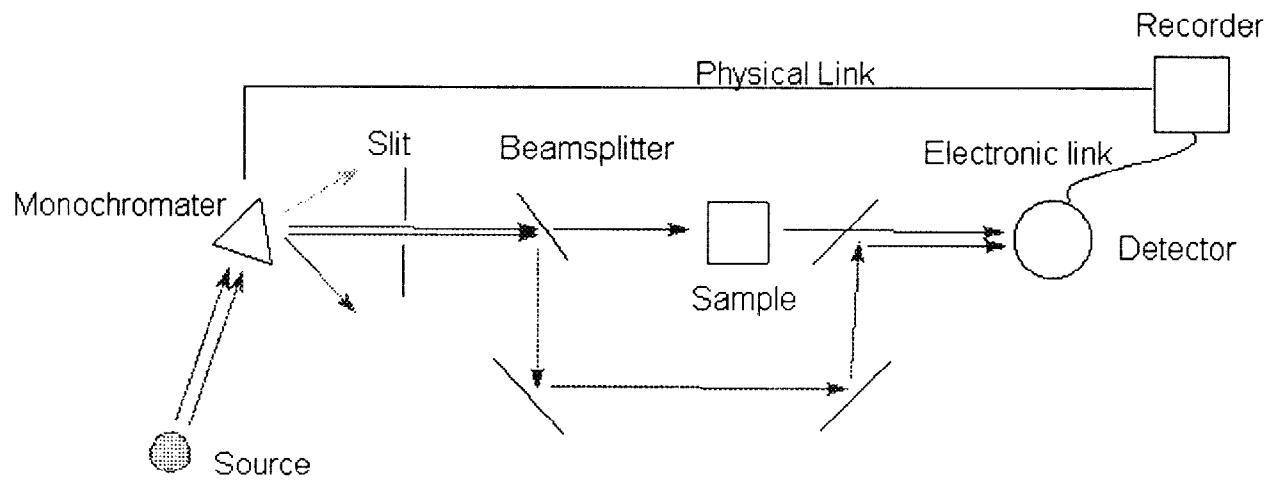
On-line MS

- Commercial instrument
- Automotive applications
- Incinerator and boiler applications
- Small-scale studies of pyrolysis
- On-line meas.
- Fast, 0.05 ppb Benzene, 2 sec.
- Broad range of species, 0 – 500 a.m.u
- Expensive

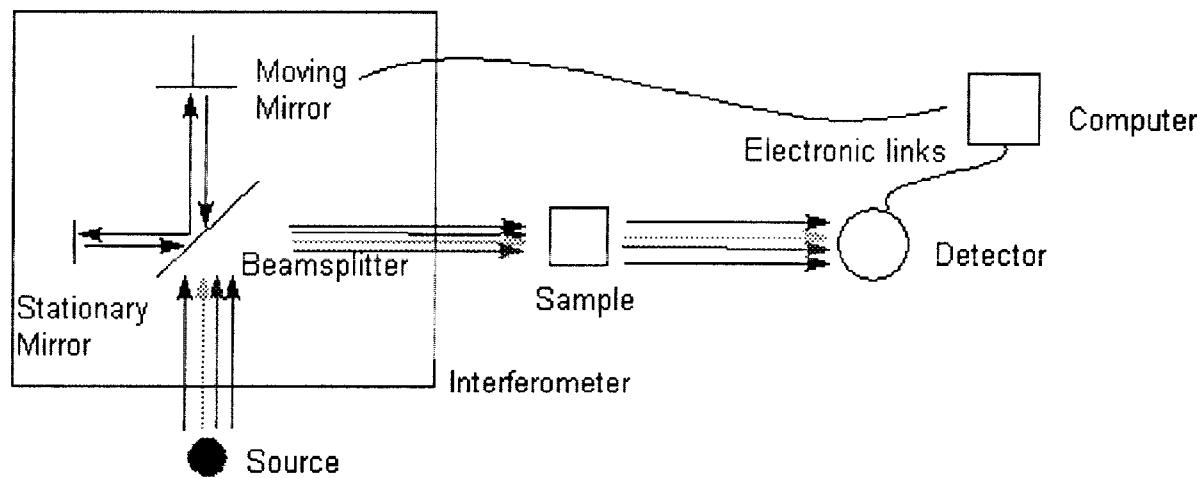
Fourier Transfer InfraRed (FTIR) spectrometry

- Several gases simultaneously
- Fast measurements
- High optical throughput
- Low limits of detection
- Stable calibration





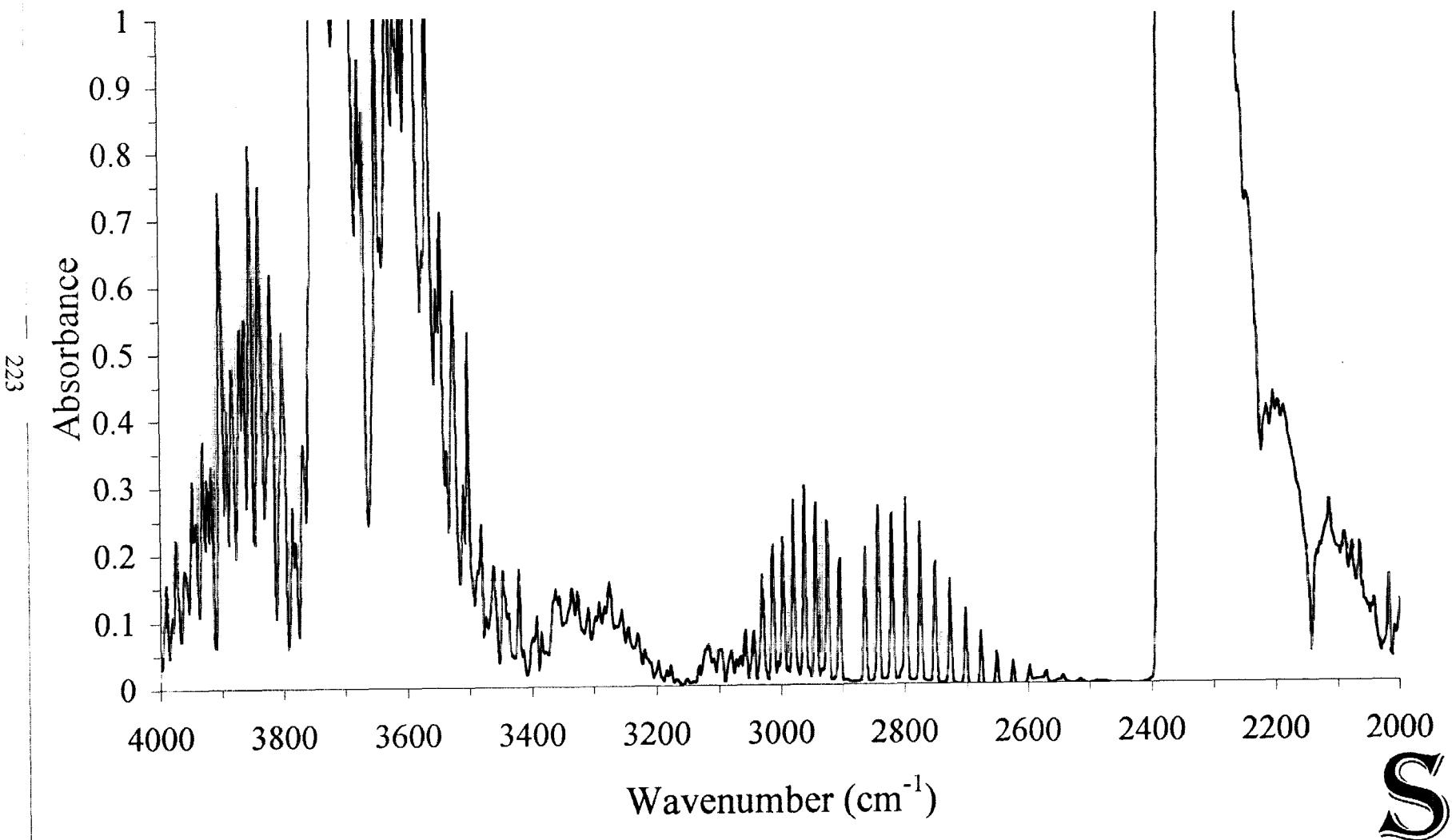
Classical
spectrometer



FTIR
spectrometer

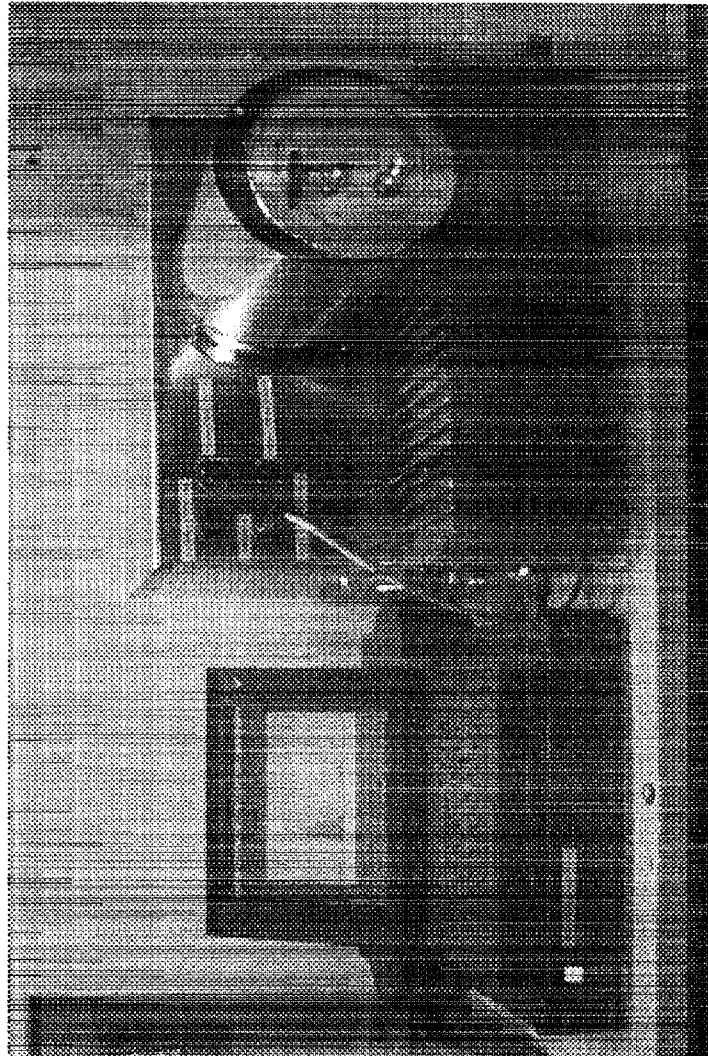
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FTIR Spectrum



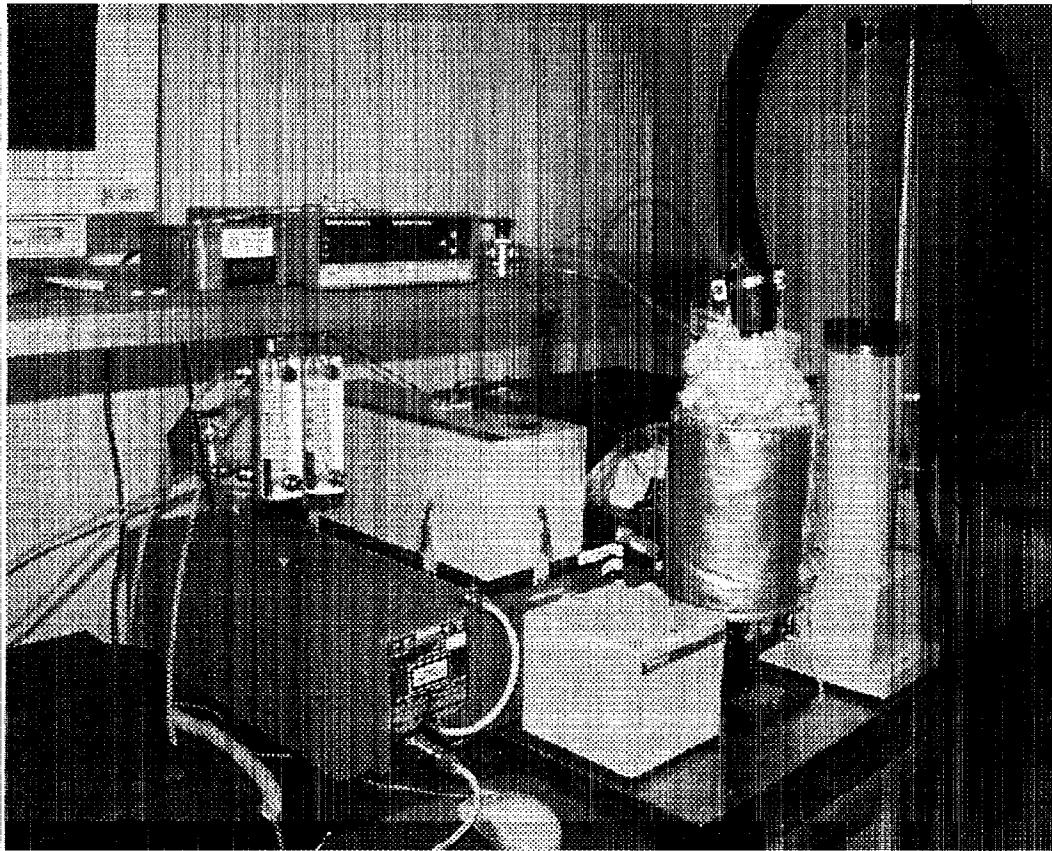
The FTIR apparatus

- IR source
- Interferometer
- Optics
- Gas cell
- Computer



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Well-equipped FTIR



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- Fast scanning interferometer
- 2 cells, one "pre-calibrated"
- 2 detectors

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Temporal resolution and detection limits I

Detection limit: Path-length, Signal-to-noise ratio

Signal-to-noise ratio:

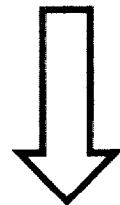
- Number of scans (**temporal resolution**)
- The Spectral resolution
- Optical components
- Quality of detector

Increased temporal resolution  **poorer detection limits**

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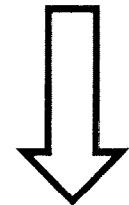
Temporal resolution and detection limits II

Time



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S/N



Number of scan	Collect. time (s)	DTGS	MCT
		MDL (ppm)	MDL (ppm)
1	1-3	8	2
2	2-6	5.6	1.4
4	4-12	4	1
8	8-24	2.8	0.7

MDL: Minimum Detection Limit, in example for HCl

Spectral resolution: 4 cm⁻¹, Path-length: 4.8 m

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Example of detection limits (MDL)

Gas	MDL (ppm)
CO	7
NO	8
NO ₂	0.5
SO ₂	1
HCN	0.5
HCl	5
HBr	10
HF	2

- Path-length: 4.8 m
- No. of scans: 3 (9 sec.)
- Detector: DTGS
- Resolution: 4 cm⁻¹

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